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increased, tano decreased. Samples with low post-curing times showed higher magnitudes of tano than samples with longer post-curing time. IPN foams

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Interim Technical Report

ENERGY ABSORPTION OF POLYURETHANE BASED POLYMER ALLOYS

GRANT NUMBER: DAAG29-85-K-0129

Polymer Institute University of Detroit January 31, 1986 Bijan Sedghi Kachorn Wongkamolsesh Daniel Klempner Kurt C. Frisch



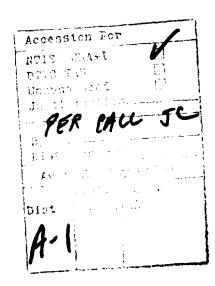


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INTRODUCTION AND OBJECTIVES

The objectives of this study are to develop interpenetrating polymer network (IPN) elastomers and foams which exhibit good sound attenuation characteristics over a broad frequency and temperature range. In this study the effect of various fillers with platelet geometry, such as graphite and mica, were investigated. The platelet geometry of the fillers is believed to encourage energy dissipation via shear contact of the planar particles. The fillers were incorporated into IPN elastomers by random batch-mixing and rubber milling. The effect of post-curing on the dynamic mechanical properties $(\tan \frac{1}{2})$ of IPN elastomers was also studied. IPN foams with different polyurethane/epoxy ratios were prepared and the sound absorption was measured by the impedance tube method.

II. EXPERIMENTAL

A. Materials

The materials used in this study are summarized in Table I. The polyols, chain extender, and epoxy resin were degassed under vacuum at 70° C for 24 hours. The other chemicals were used as received from the manufacturers.

B. Preparation of Samples

1. IPN Elastomers

The IPN elastomers based on polyurethane and epoxy were prepared by the simultaneous polymerization technique. One component contained the isocyanate (Isonate 143L) and epoxy resin (DER 330). The other component contained polyols (Niax 31-28), chain extender (Isonol 100), urethane catalyst

(T-12), epoxy catalyst (BF_3 -etherate) and various fillers.

The two components were mixed together for one minute at room temperature using a high speed mechanical stirrer. The mixture was then poured into a pre-heated mold and pressed on a laboratory platen press at 100° C. The samples were then removed from the press (after curing for 30 min.) and then post cured in an oven at 100° C for 16 hours. Samples were conditioned at 25° C and 50% relative humidity for at least three days prior to testing.

2. IPN Foams

IPN foams were prepared by the one shot, free rise method. Since difficulties were encountered in previous studies due to the slow reaction of the epoxy component at ambient temperature, the mixing temperature in this study was raised to 80° C. The Niax polyol 31-28 and epoxy resin DER 330 were first preheated to 80° C. The surfactants (DC-193, L-540), blowing agents (water, Freon 11A), chain extender (Isonol 100), polyurethane catalyst (T-12, A-1), and catalysts for epoxy resin (XU-213, DMP-30) were added and thoroughly mixed with a high speed stirrer for two minutes. The cream time, rise time and tack-free time were recorded. The foams were cured at 90° C for 16 hours and conditioned at 25° C and 50% relative humidity for at least three days prior to testing.

C. <u>Testing</u>

Dynamic Mechanical Spectroscopy

All dynamic mechanical measurements were conducted on a Rheovibron dynamic viscoelastometer, DDV II (Toyo Manufacturing Co.) at a scanning rate of 1 to 2° C per minute in the glass transition region or every 3 to 5° C per minute in the non-transition region. The specimens were in the form of rectangular films with dimensions of 2 cm in length, 0.1 cm in width, and

 $0.05~\rm cm$ in thickness. The specimens were inserted into the chamber and cooled to $-50^{\rm O}{\rm C}$ where the measurement began. All tests were carried out at a frequency of 110 Hz.

Standing Wave Apparatus (Bruel & Kjaer, Type 4002)
 For Sound Absorption Measurement

This apparatus (Type 4002) is designed for easy and quick determination of the absorption coefficient of acoustic materials by the standing wave method. The advantages of the method are that only small circular samples, about 10 cm in diameter, are needed. The principal of the measurement method is shown in Figure A. The loudspeaker at one end of the tube is operated at the desired test frequency from an audio-frequency oscillator with 6 ohms output impedance and a distortion of less than 1% (B.F. Oscillator Type 1022). The sound waves move through the tube and strike the sample which is placed in a sample holder with a thick back plate, to avoid all sound absorption by the apparatus itself. The sound waves are then partly reflected at the sample. The resultant of the incident wave with amplitude 1 and reflected wave with amplitude r is a standing wave pattern with alternate sound maxima 1+r and minima 1-r in the tube. From the ratio n of these sound pressure maxima and minima the reflection coefficient r (see equation below) follows directly.

$$r = \frac{n-1}{n+1} \tag{1}$$

However, we are interested in the absorption coefficient α , i.e. the ratio of the energy absorbed by the sample to the incident energy. In other words, $\alpha = 1 - r^2$, from which, with the aid of the relation (1):

$$\alpha = \frac{4}{n + \frac{1}{n} + 2} \tag{2}$$

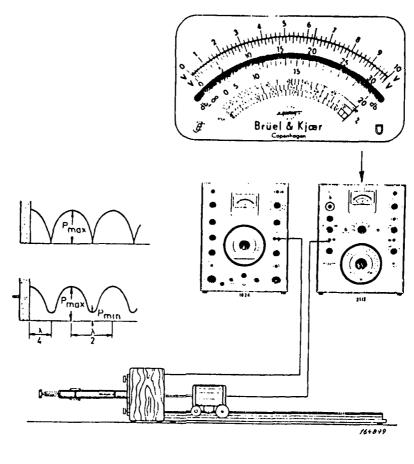




Figure A. The Standing Wave Apparatus Type 4002 mounted for low frequency Vs. Octave Analyzer 2112.

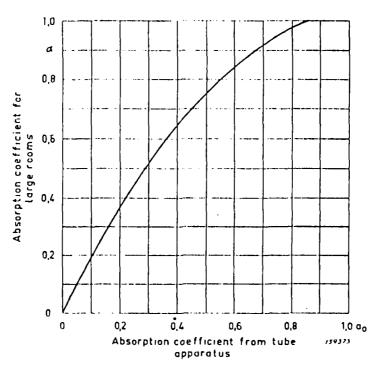


Figure B. Relation between absorption coefficient of large rooms and tube apparatus.

The sound field is explored by means of a probe microphone, movable on a track equipped with a scale on which the exact distance between the probe entrance and test sample can be read. The microphone voltage is amplified by a selective amplifier to reduce the influence of hum and noise and higher harmonics, which are inevitably generated by the speaker in the tube. Particularly suitable for this purpose is the 1/3 Octave Analyzer 2112 with 33 fixed filters from 22 Hz to 45 kHz and three scales, 0-100%, 0-70%, and 0-30%.

The absorption coefficient is determined by the tube measurement method only at normal incidence, which is why the measured coefficients are generally somewhat smaller than those determined by the reverberation room method according to W.C. Sabine's formula. In Figure B a curve is shown which indicates the relationship between the results of the tube method and the reverberation room method.

3. Density

The density of foam samples was measured according to ASTM D-1622.

III. RESULTS AND DISCUSSION

A. Effect of Fillers on IPN Elastomers

The effect of various fillers on the morphology ($\tan \delta$) of IPN elastomers was investigated. The polyurethane/epoxy IPN (formulation #43, previous Report No. 1), 60/40 with 2% Isonol 100 as chain extender, was selected as a model formulation (labelled as formulation #1 in this report). This formulation was repeated to check the reproducibility and consistency of the preparation. The results are shown in Figures 1 and 2. Comparison of both figures indicates the reproducibility of previous work.

Since it is postulated that fillers with platelet geometry shall encourage energy dissipation via shear contacts of the planar particles, fillers with platelet structure such as graphite flake and mica flake were incorporated in various proportions in the PU/epoxy IPN.

Three different particle sizes of graphite flake (#1 -20 mesh to +80 mesh, #2 -50 mesh to +200 mesh, and #3 -80 mesh down) were investigated. Initially, the graphite filler (platelets) was incorporated into the PU/epoxy IPN by random batch mixing with a high speed stirrer and then pressing on a platen press. The dynamic mechanical spectra of three graphite filled IPNs (10%) are shown in Figures 3-5 and the comparison of tan& of unfilled and graphite filled elastomers is shown in Figure 6. The result in Figure 6 shows a little increase in tan and a shift of Tg to about 10°C lower. When the amount of graphite filler was increased to 20% and 40% as shown in Figures 7 and 8, it was found that the tan magnitude decreased (Figure 9). An increase as well as a broadening was expected with this filler (studies at the University of Dayton showed these results, i.e. increase and broadening of tan& with addition of graphite platelets).

The graphite filler was also incorporated into the IPN elastomer on a rubber mill. This technique was used so that the shear force between the rolls will align the platelets of the graphite filler in the same direction. This should facilitate energy dissipation. Twenty and forty percent of #2 graphite filler were incorporated into the IPN by this technique and their dynamic mechanical spectroscopy were measured and are shown in Figures 10 and 11. At 20% graphite filler there was no change of $\tan\delta$ observed, however, at the 40% level the $\tan\delta$ breadth decreased.

In the case of mica filler, the filler was incorporated into the IPN system with and without Isonol-100 chain extender. The same phenomenon was

was observed. As the amount of filler increased, the $tan\delta$ magnitude decreased (Figures 12 and 13). In the system with Isonol-100 chain extender, Figure 14, the $tan\delta$ did not show significant change.

Figures 15 and 16 show the dynamic mechanical spectra of the Dicaperl FP-1010 (hollow glass bubble filler) and Sundex 740T plasticizer filled IPN elastomers. At the 10% level, in both cases, there was no change of $\tan\delta$ observed. The amount of Dicaperl filler and plasticizer will be increased in the next study.

B. The Effect of Post-Curing Time

The effect of post-curing was investigated by measuring the dynamic mechanical properties of the IPN elastomers after the samples were post-cured for different periods of time (e.g. 0, 2, 8, and 16 hours). The results are shown in Figures 17-20 and the $tan \delta$'s are compared in Figure 21. It was found that with no post-curing, the sample showed a high tano However, as the postcuring time increased to 2, 8 and 16 hours, the $tan\delta$ magnitude decreased with the Tg shifted to higher temperatures as expected ($\sim 10^{\circ}$ C). The high tan δ of the sample without post-curing or low post-curing time was probably due to the uncured epoxy as well as the polyurethane. The molecular chains are partially loosening and the small chain segments can move. An attempt was made to stabilize the uncured condition by immersing the uncured sample in methanol (to "kill" the reactive epoxy groups) for 3 hours and drying the sample under vacuum for 24 hours. The dynamic mechanical spectroscopy was taken and is shown in Figure 22. The $tan\delta$ was found to decrease to the same level as the sample post-cured for 8 hours. This area will be further investigated.

C. Foams

The IPN foams were prepared by mixing the preheated polyol and epoxy resin at 80°C. The high temperature was required in order to speed up the epoxy reaction. However, at this temperature, the evaporation rate of the blowing agent, Freon 11A, was too rapid and thus a second blowing agent, water, was necessary in order to obtain low density foams. Using water in the formulations required adjusting the amount of the catalysts, since problems with collapsing and shrinkage arose. It was observed that this foam system was sensitive to the amount of catalyst used. Efforts were made to formulate some samples by changing the percent water used and adjusting the amount of the catalysts for different polyurethane/epoxy (PU/epoxy) ratios.

IPN foams were prepared with PU/epoxy ratios of 70/30, 60/40, 50/50, 40/60, and 30/70. An isocyanate index of 105 was used in preparing these samples, Figures 23-28 show the percentage of sound absorption of foam containing 2% Isonol 100 as chain extender. In these formulations the urethane component contained excess Isonate 143L to react with the pendant hydroxyl group of the epoxy represent the sound absorption of the foams without DER 330. Figures 29-31 Isonol 100 and excluding the excess Isonate 143L to react with the epoxy. Figures 23 and 24 show the tested specimens cut out from the top and bottom of the prepared samples with a PU/epoxy ratio of 70/30. This was done to investigate how homogeneous the samples were in the direction of foam rise. It was suspected that concentration of the epoxy may tend to be more at the bottom of the prepared samples, since the unreacted epoxy may descend due to gravity. This phenomenon is more likely to happen while the foam is rising and gelling. Therefore, it is important to have balanced rates of reactions of both the polyurethane and the epoxy systems. The lower portion of the sample, Figure 24. shows about 10-15% lower sound absorption than the upper portion, Figure 23, at higher frequencies.

This, as mentioned above, could be due to more available hard segments (epoxy crosslinks) in the lower portion of the sample. In Figure 32, the polyurethane reaction was slowed down by applying less amounts of catalysts, A-I and T-I2.

The curing effect on the percent sound absorption of a sample with a PU/ epoxy ratio of 40/60 is shown in Figures 33 and 34. As can be seen from these figures, the cured specimen has a lower sound absorption capability over the test frequencies. This is most likely due to uncured epoxy cortaining a distribution of MW and therefore broader energy absorbing abilities (wider Temp. and frequency range). Figures 35 and 36 represent foams without the blowing agent, Freon 11A, at different levels of catalyst, DMP-30. The excess amount of DMP-30 promoted trimerization of Isonate 143L and gave a harder structure to the foam which resulted in lower percent sound absorption. Foams were prepared with a different type of blowing agent (dichloromethane) which has a higher boiling point than Freon 11A. Samples made with blowing agent from preheated (also not preheated) epoxy and Niax polyol show lower percent sound absorption. The test results for both preheated and not preheated samples are shown in Figures 37 and 38. The sample tested in Figure 28, due to closed and tight cell structure, showed poor sound absorbing properties. This demonstrates the important effect of cell structure of a foam on dissipation of energy and sound.

IV. FUTURE WORK

- A. Different fillers and amounts of fillers and plasticizer will be further investigated.
- B. Various types of fillers will be incorporated into the IPN foam and the sound attenuation properties will be studied.
 - C. Three-component IPNs will be studied.

Table I Materials

Materials	Chemical Composition	Eq.Wt.	Supplier
Isonate 143L	Carbodiimide modified diphenylmethane diisocyanate	143	Dow Chem. Co.
Niax 31-28	Graft copolymer of poly(oxy- propylene)(oxyethylene) adduct of glycerol	2004.5	Union Carbide
Mondur MR	Polymeric isocyanate	133.3	Mobay
Isonol 100	N,N'-Bis(2-hydroxypropyl)aniline	104.5	Dow Chem. Co.
DER 330	Bisphenol A-Epichlorohydrin epoxy resin	177-178	Dow Chem. Co.
T-12	Dibutyltin dilaurate	-	M & T Chem.
Niax A-1	70% Bis(2-dimethylaminoethyl) ether solution in dipropylene glycol		Union Carbide
BF ₃ (0 ₂ CH ₅) ₂	Boron trifulorine etherate		Eastman Chem.
DMP-30	2,4,6-Tris (dimethylaminomethyl) phenol		Rohm & Haas
XU-213	BC1 ₃ -Amine complex		Ciba-Geigy
Freon 11A	Trichlorofluoromethane		E.I. duPont
DC 193	Silicone copolymer surfactant		Dow Corning
L-540	Silicone surfactant	-	Union Carbide
Suzorite mica	Mica flake	-	Marietta Resources Inter. Ltd.
Dicaperl FP1010	Hollow glass bubble filler	-	Grefco Inc.
Sundex 740T	Plasticizer		Sun Products
Graphite flake #1	Size: -20 Mesh + 80 Mesh		Asbury Graphite Mills, Inc.
Graphite flake #2	Size: -50 Mesh + 200 Mesh		Asbury Graphite Mills, Inc.
Graphite flake #3	Size: -80 Mesh down		Asbury Graphite Mills, Inc.

#1 Graphite, g #2 Graphite, g #3 Graphite, g #3 Graphite, g Mica, g Dicaper1 FP1010, g Sundex 7401, g	Epoxy DER 330, g BF ₃ -etherate, g PU/Epoxy ratio	Polyurethane Isonate 143L, g Niax 31-28, g Isonol 100, g T-12, g	Figure No. Sample No.
	28 0.6 60/40	3.91 37.34 0.76 0.06	
1111	24.8 0.6 60/40	4.31 33.08 0.68 0.06	1 2
7	28 0.6 60/40	3.91 37.34 0.76 0.06	~ ω
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7	28 0.6 60/40	3.91 37.34 0.76 0.06	72 4
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1 1 4 1 1 1	28 0.6 60/40	3.91 37.34 0.76 0.06	14
. 7	28 0.6 60/40	3.91 37.34 0.76 0.06	15 12
71111	28 0.6 60/40	3.91 37.34 0.76 0.06	16 13
	28 0.6 60/40	3.91 37.34 0.76 0.06	17**

^{*}Fillers are incorporated by rubber mill technique **Figures 17-22 have the same formulation

Table III Formulations for IPN Foams

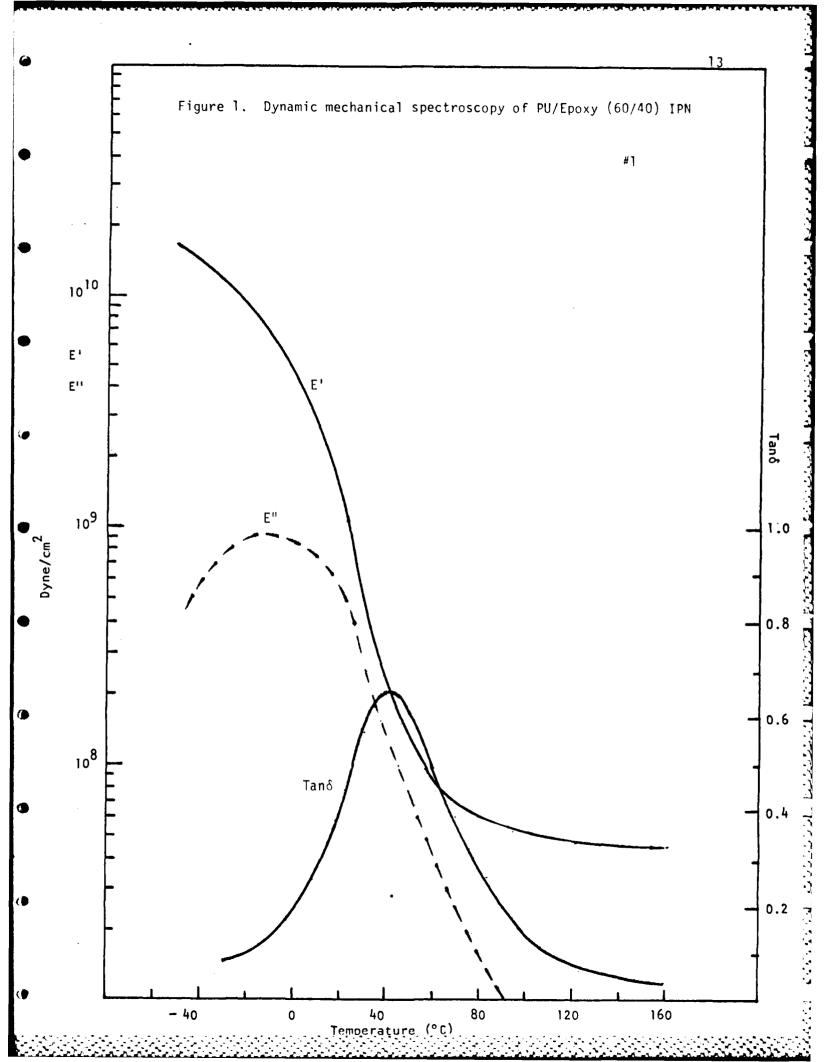
Cream time, sec. Rise time, sec Tack free time	Density, pcf	DMP-30, %	(Niax + Isonol) XU-213. %	Water/OH	Iso. Index (excess)	Isonol 100, %	PII/Fnoxv	Dichloromethane	Erson 11A	DMP-30	XU-213	DER 330	L-540	DC-193	A-1	T-12	Water	Isonol 100	Niax 31-28	Isonate 143L	Formulation No.	Figure No.
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15 180 over 1/	(3.69)																	1.43			2)	#24
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15 180	5.92	0.47	1.17	ഗ	105	2	40/60	Ġ	بر م	0.48	1.20	102.5	<u>-</u> .0	1.0	0.08	0.09	1.22	0.79	38.6	27.9	#5	#27
15 180		0.47	1.17	ഗ	105	2	30/70	ć	<u>ب</u>	0.58	1.40	119.5	1.0	1.0	0.06	0.09	0.89	0.57	28	22	#6	#28
30 250	3.5	0.56			105***		70/30	ć	ي د	0.24	0.6	42.8	1.0	1.0	0.04	0.02	1.4	•	70	28.6	#7	#29
30 250	3.93	0.56	1.4	4.5	105***		60/40	ć	بر 2	0.32	0.8	57.1	1.0	1.0	0.04	0.02	1.2	•	60	24.5	#8	#30
30 250	5.31	0.56			105***		50/50	S	3	0.40	 0	71.4	1.0	1.0	0.04	0.02	1.0	•	50	20.4	#9	#31
20 200	6.60	0.47	1.17	5	105	2,00	40/60	ć	<u>ئ</u>	0.48	 .>	102.5	1.0	1.0	0.01	0.01	1.22	0.79	38.6	27.9	#10	#32
15 190	4.55 (4.87) 7.33	2	ယ	5	105	2	40/60	Ċ	بر ح	2.0	3.0	102.5	1.0	1.0	0.02	0.01	1.22	0.79	38.6	27.9	#11 (#12)*	#33 #34
10 170	7) 7.33		1.17				-															
15 150	6.37	1.17	1.17	5	105	2,33	50/50	c	o ;	1.0	7.0	85.5	1.0	٥. ١	0.10	0.10	1.55	1.0	49	34	#14	#36
15	9.39	ω	4	6.	10	2,	50/	မှ)	2.	ω.	93			0.	0.	2.	_	49	47	#15** (#16)	#37
15 190	(6.13)			6	5	;	50	-		81	75	. 7	0	0	02	04	0	0		.7	(#16)	#38

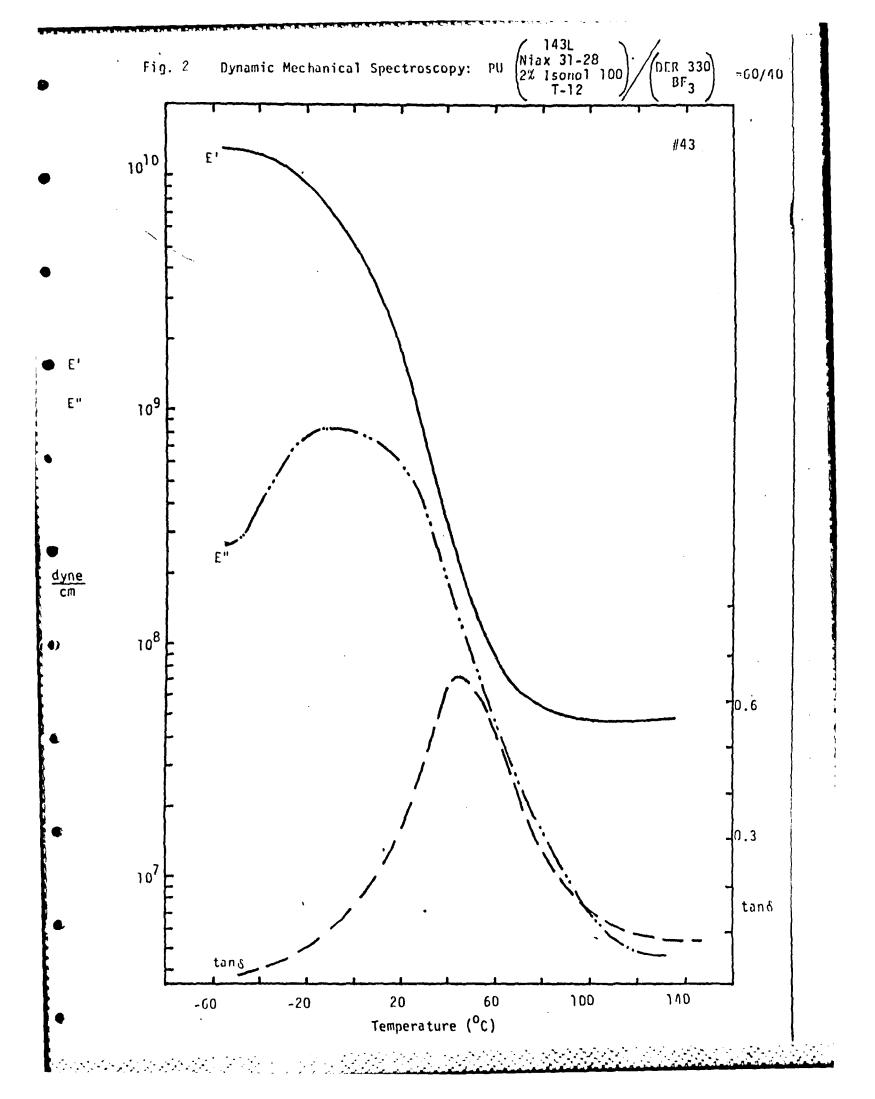
*#12 is not cured

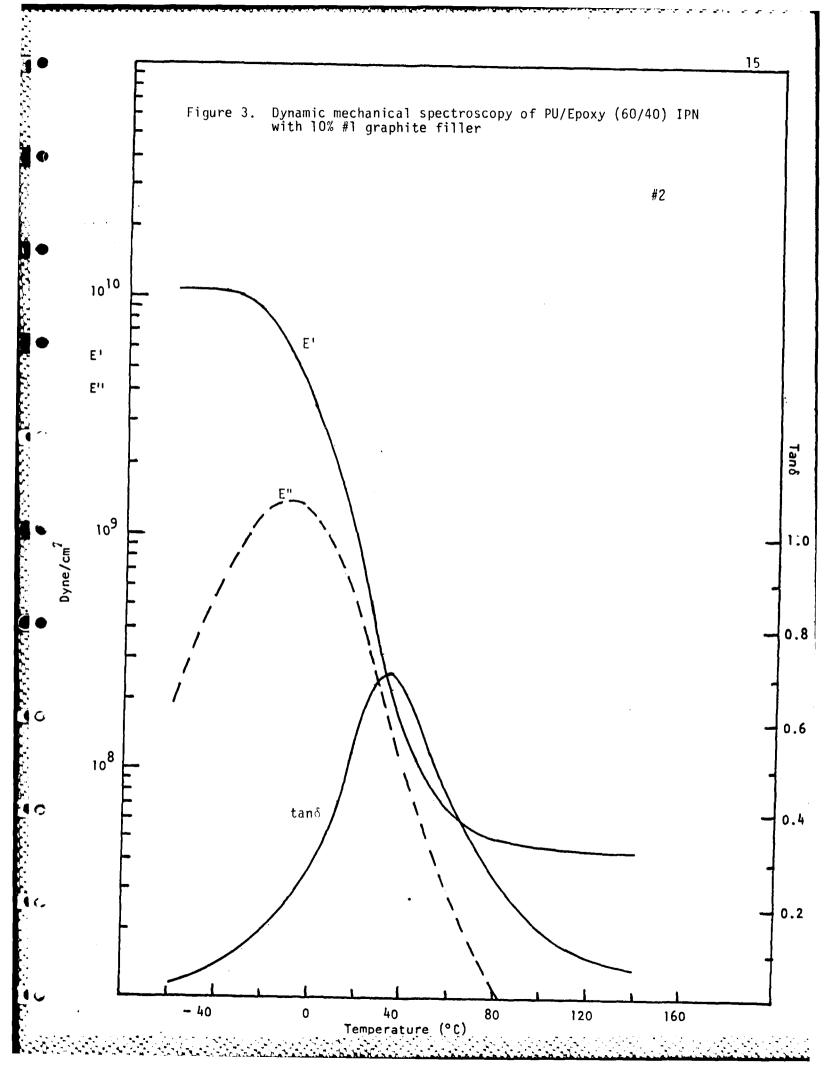
**#15 is made by hot mold method

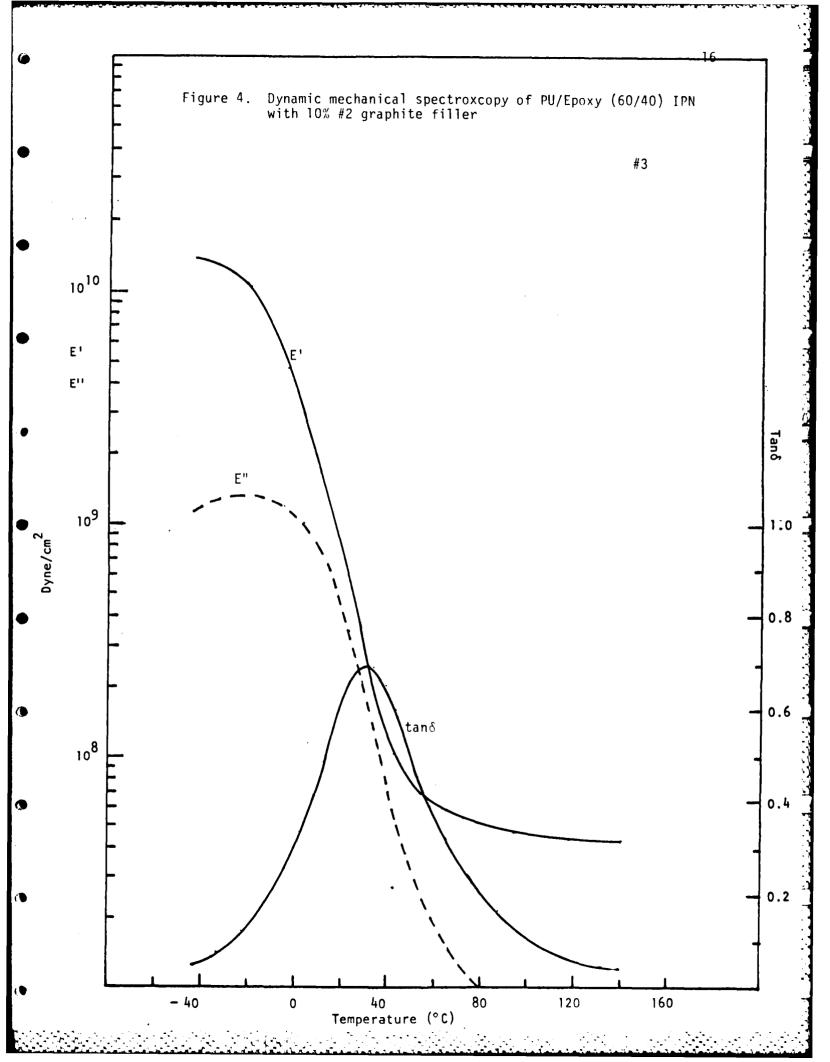
***#7, #8, #9 not excessed isocyanate index

のからは、「他のことは、ことなる」とのなどのでは、「いっこ









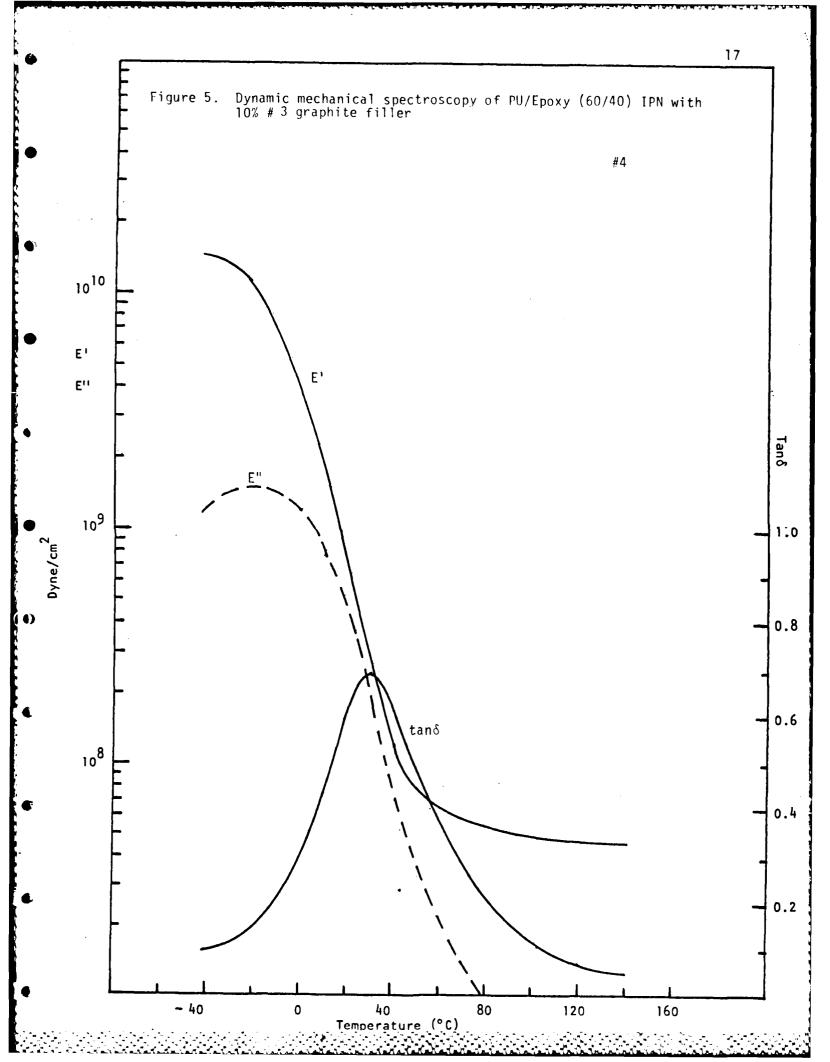
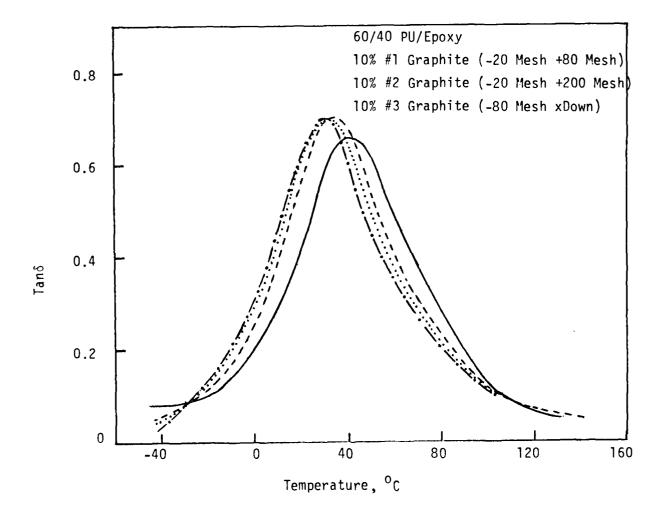
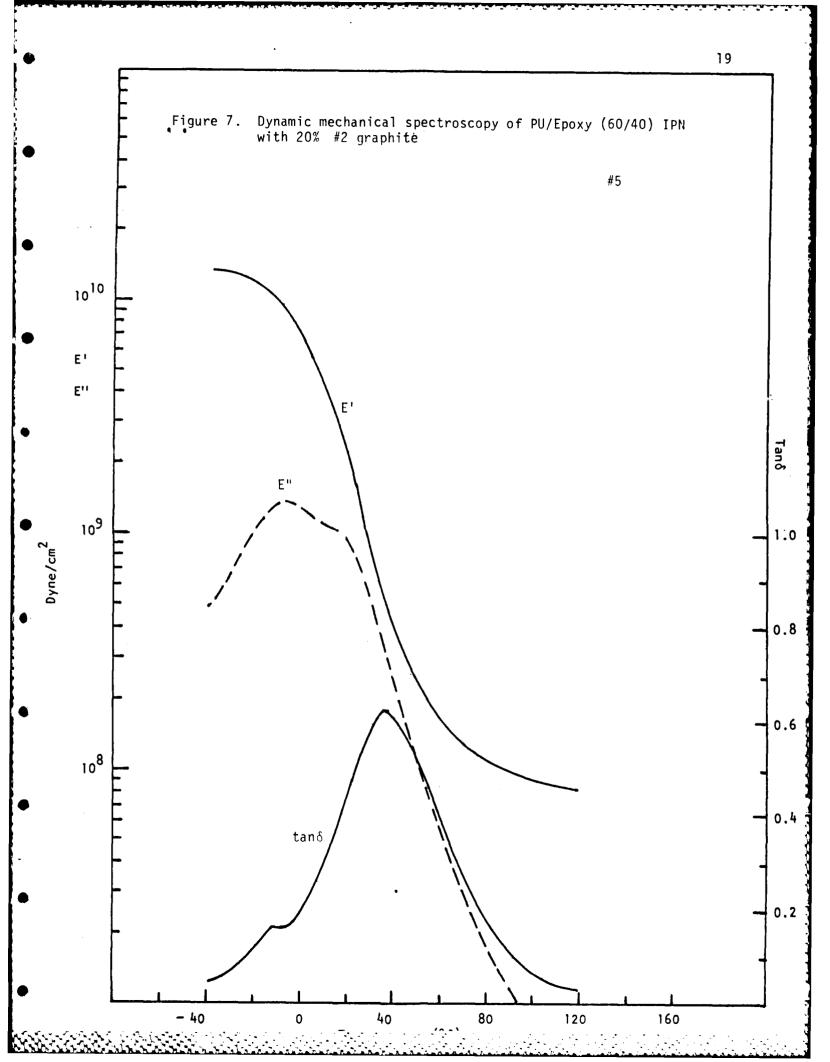


Figure 6. Comparison of the $\tan\delta$ of unfilled and graphite filled PU/Epoxy (60/40) IPN





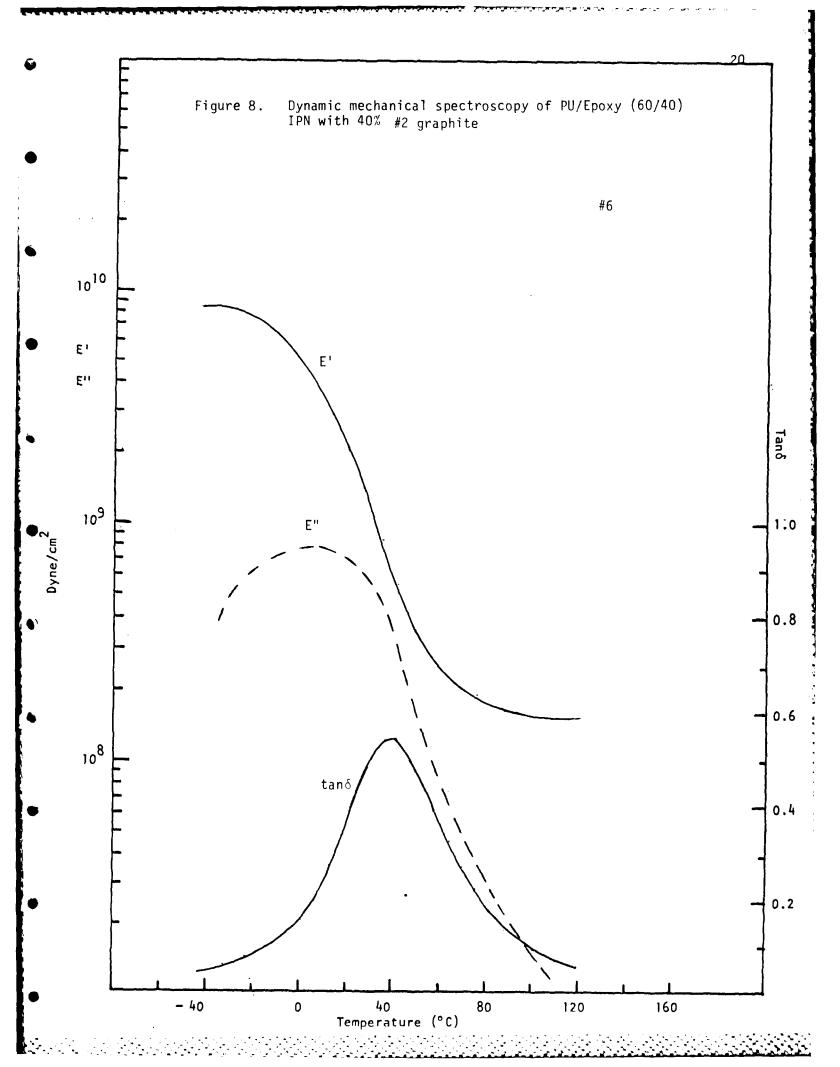
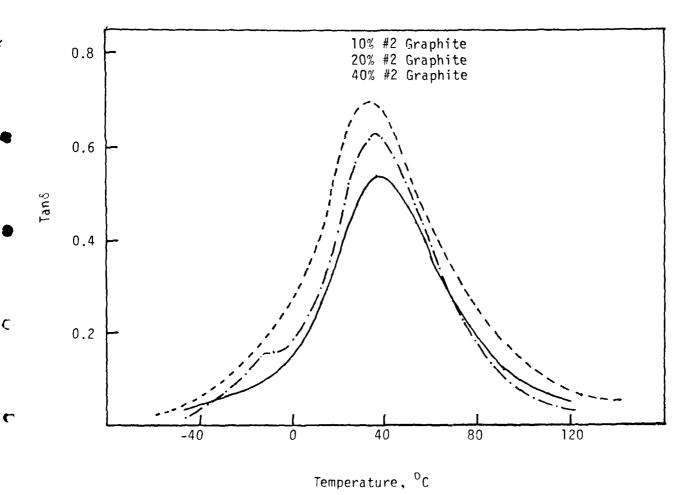
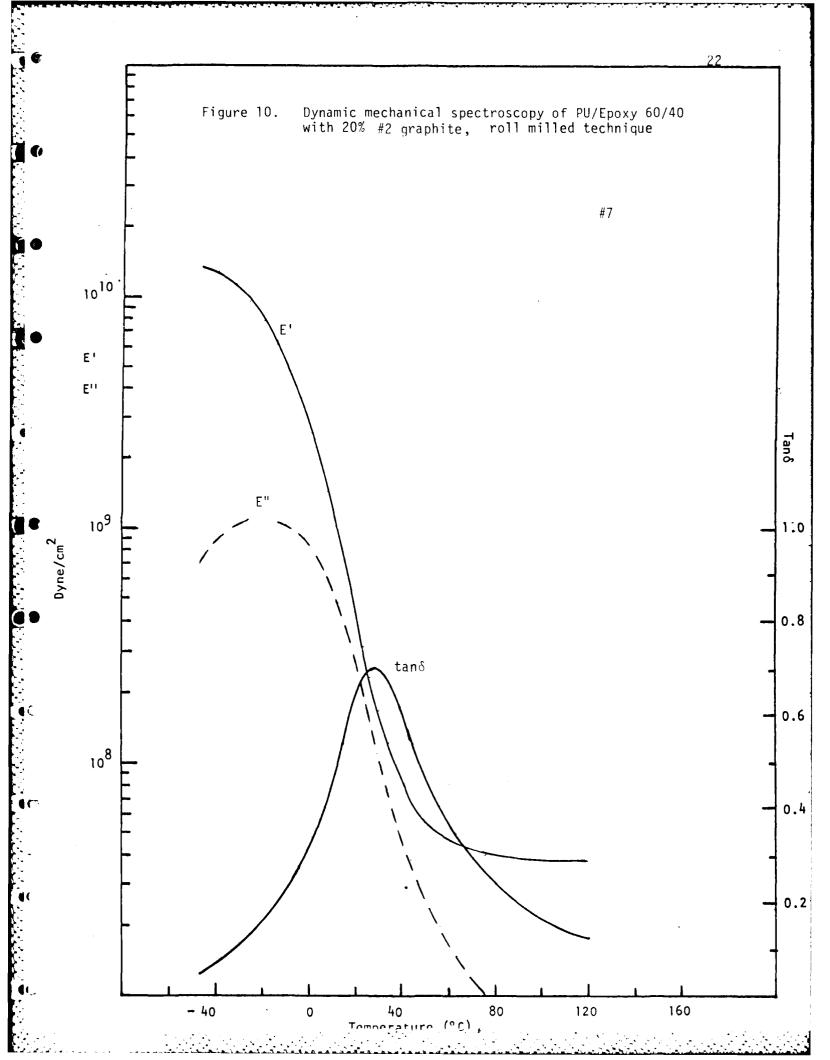
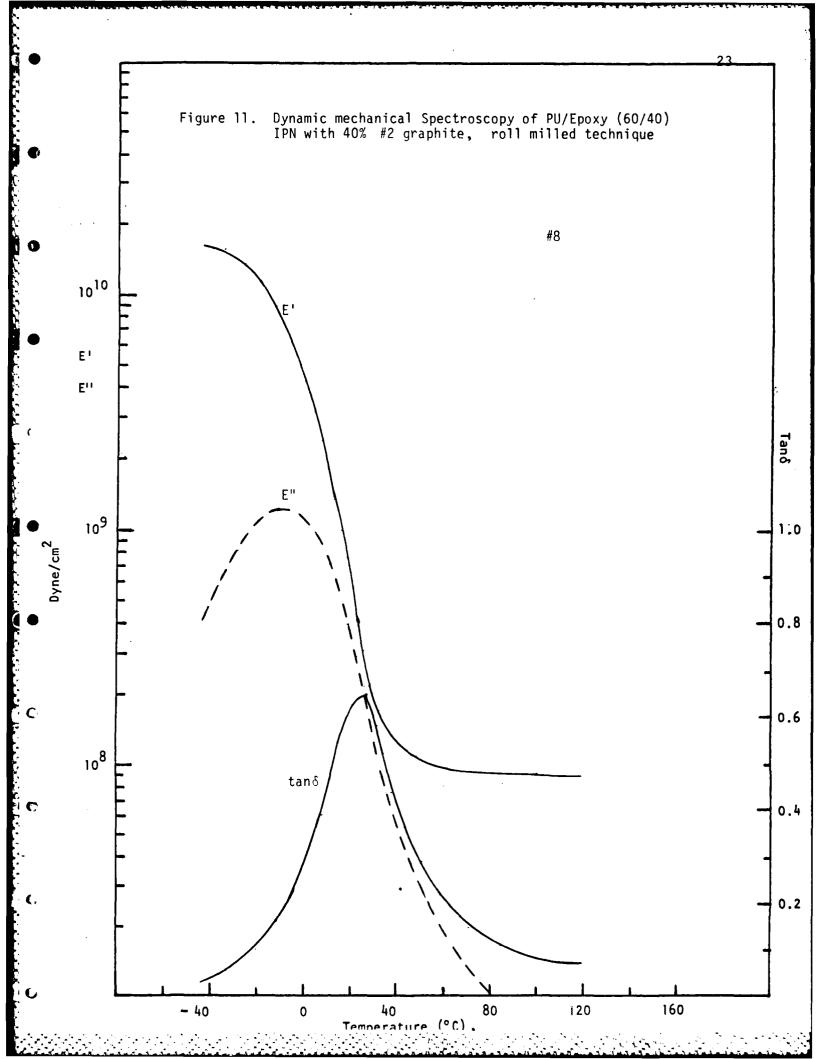
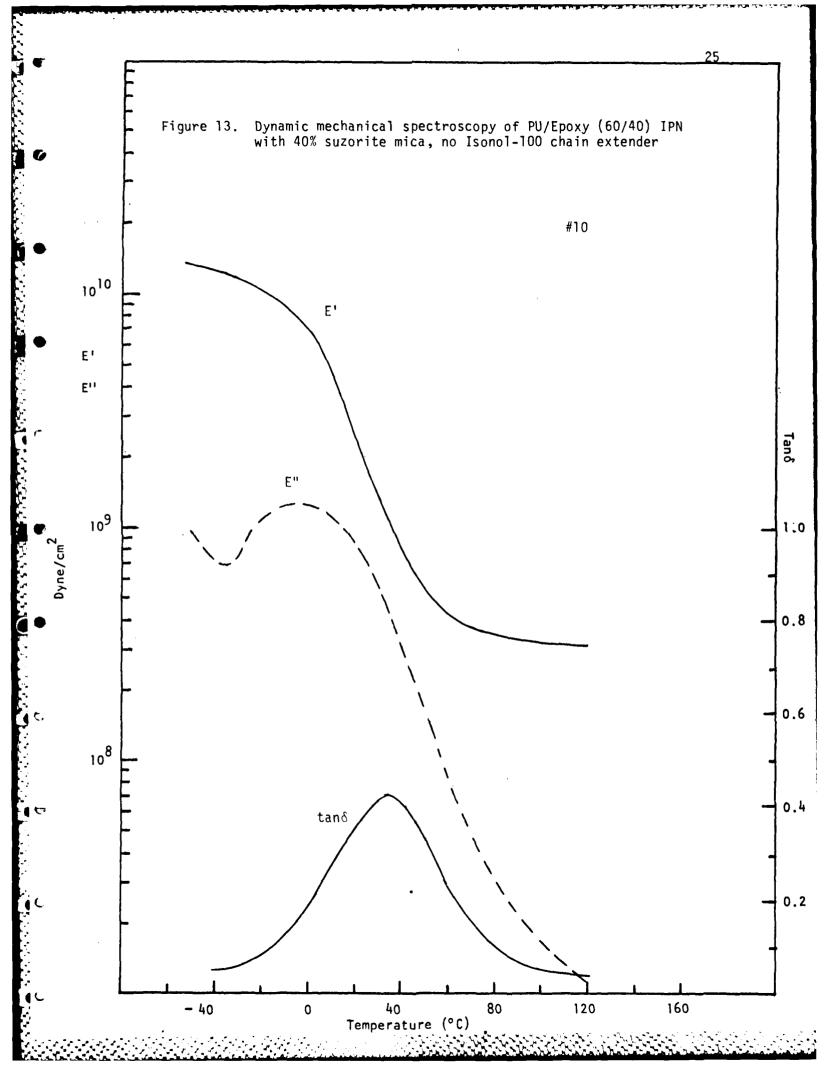


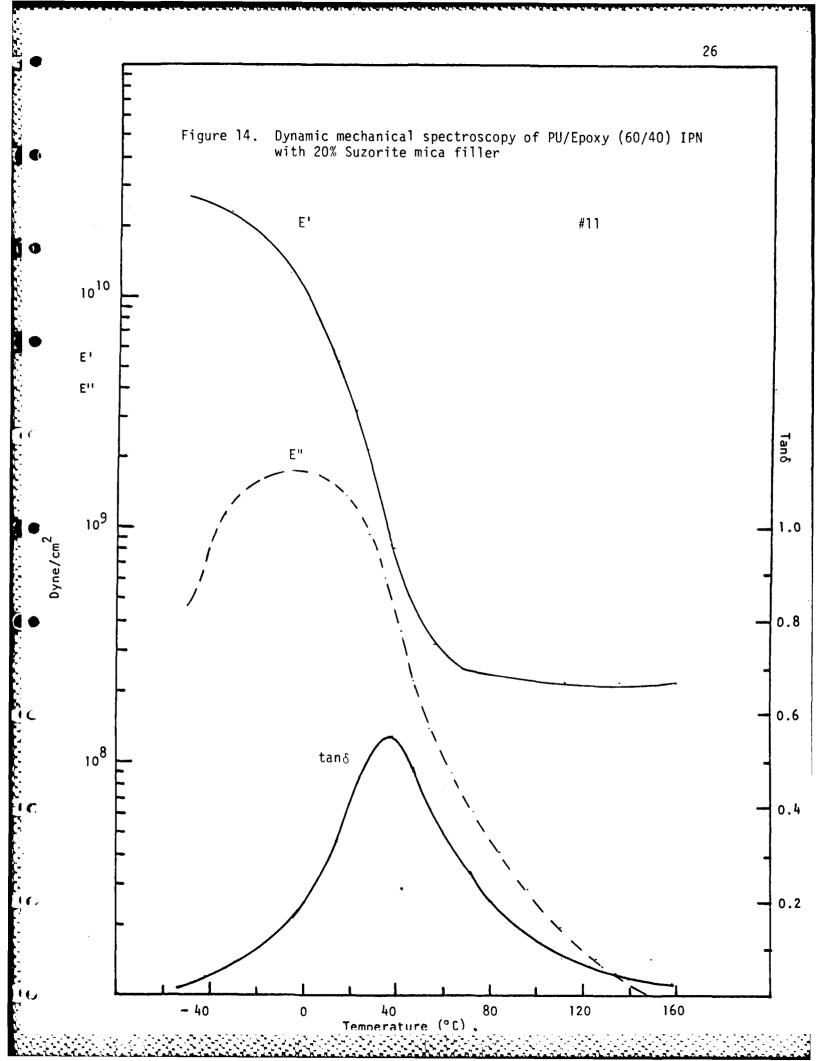
Figure 9. Comparison of the tan δ of graphite filled PU/Epoxy (60/40) IPN

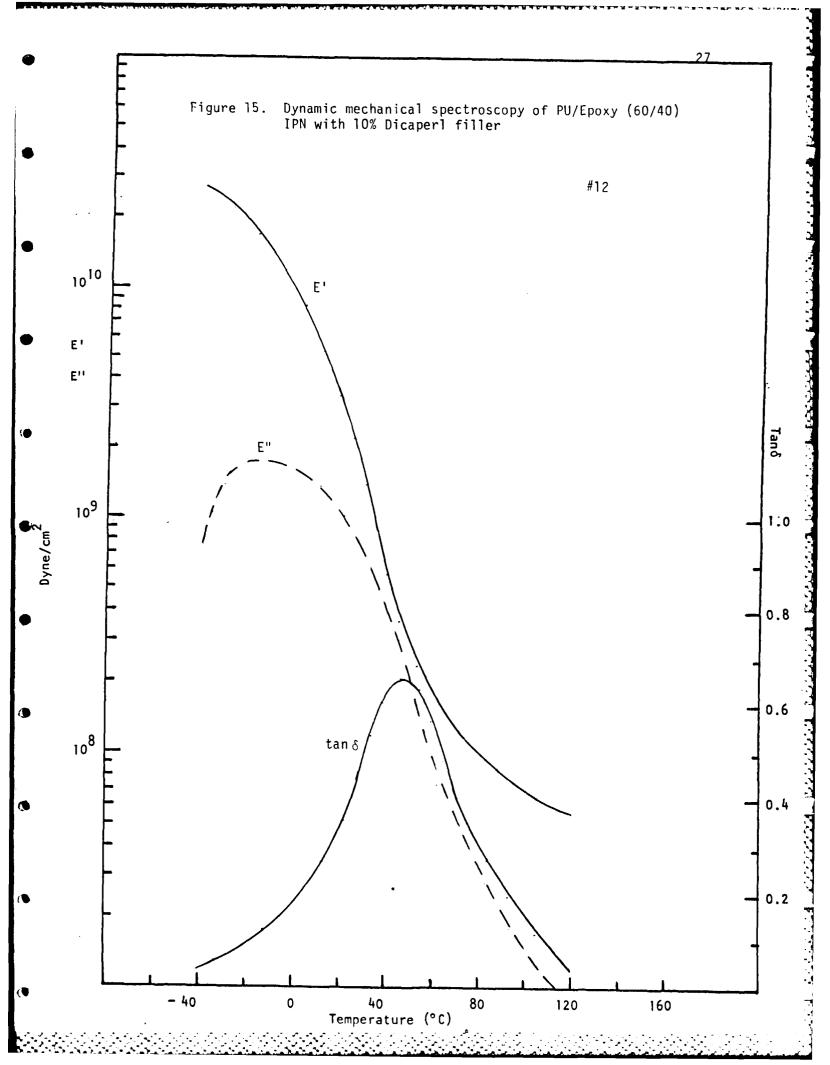


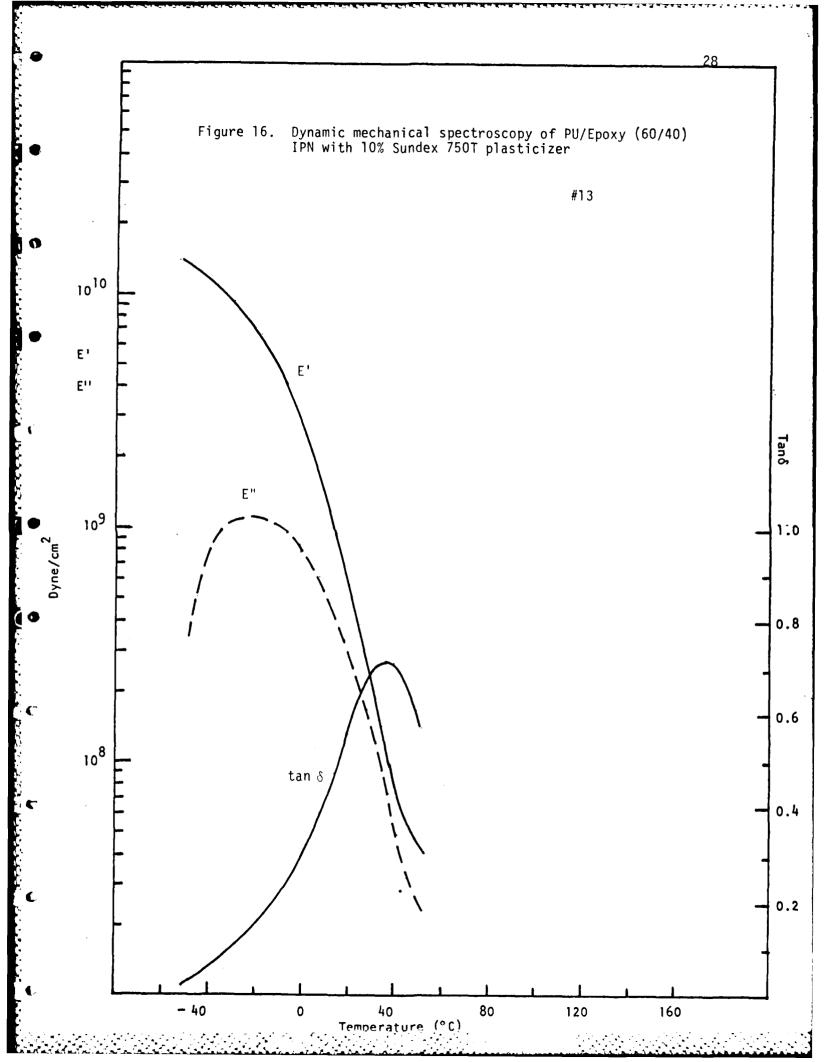


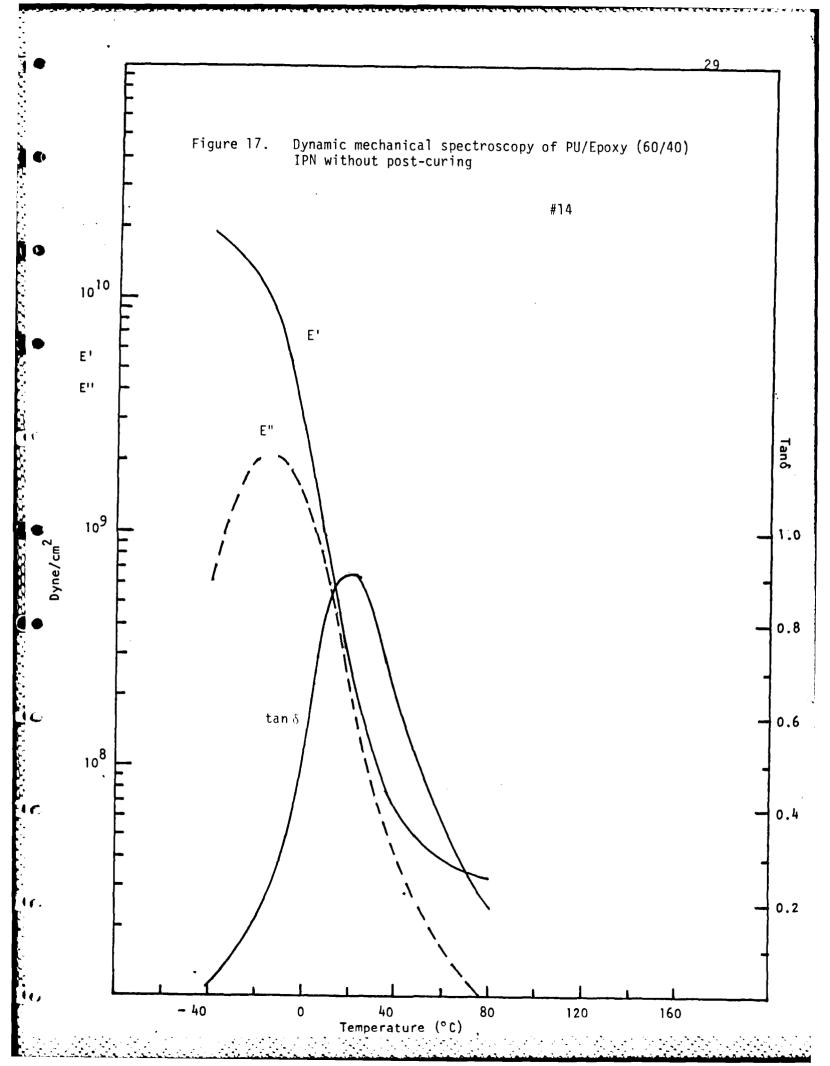


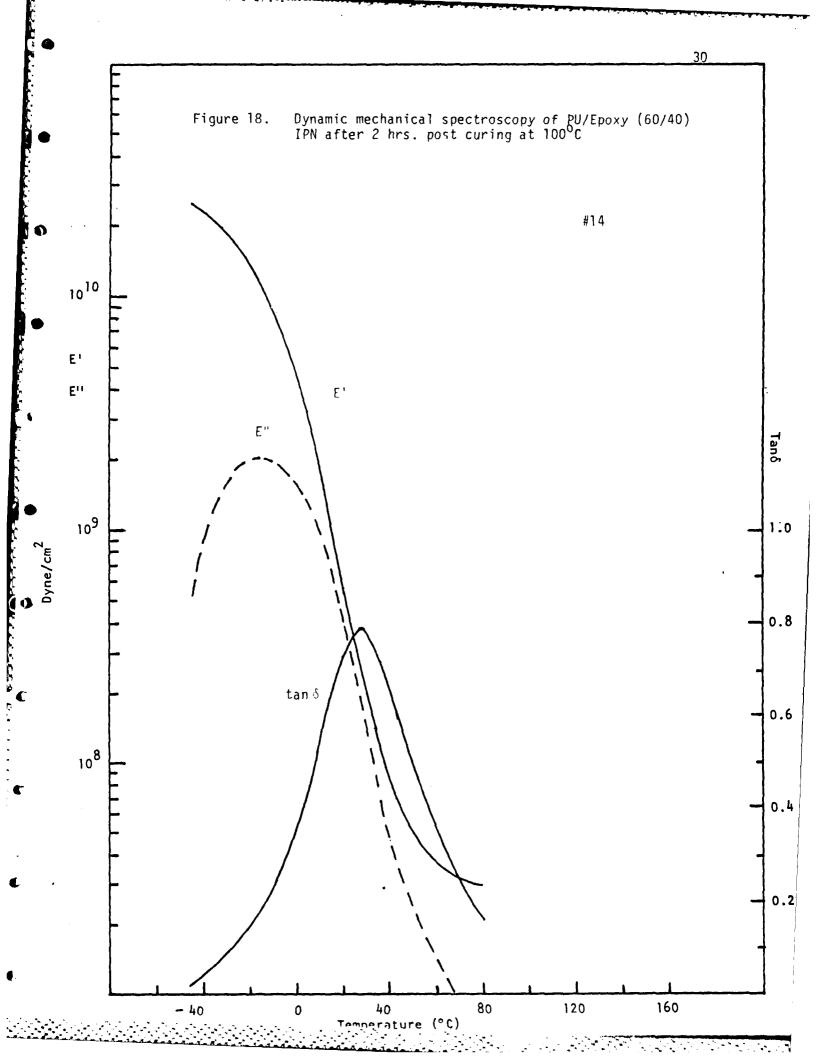












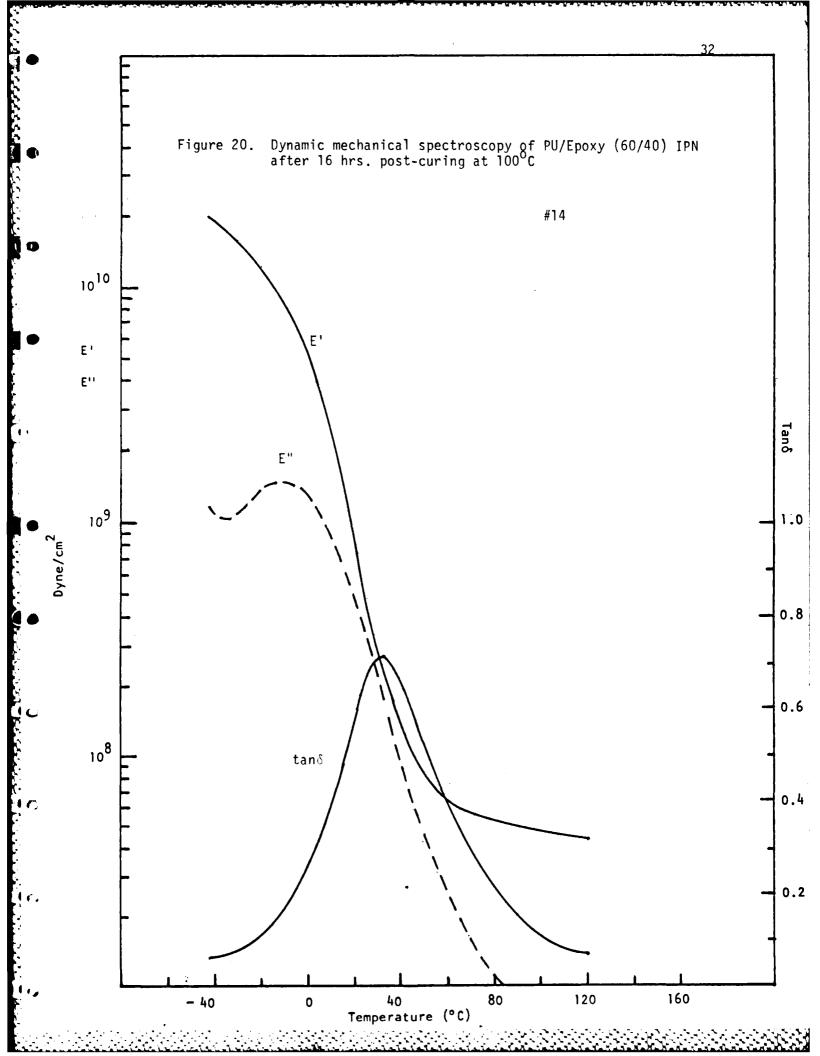
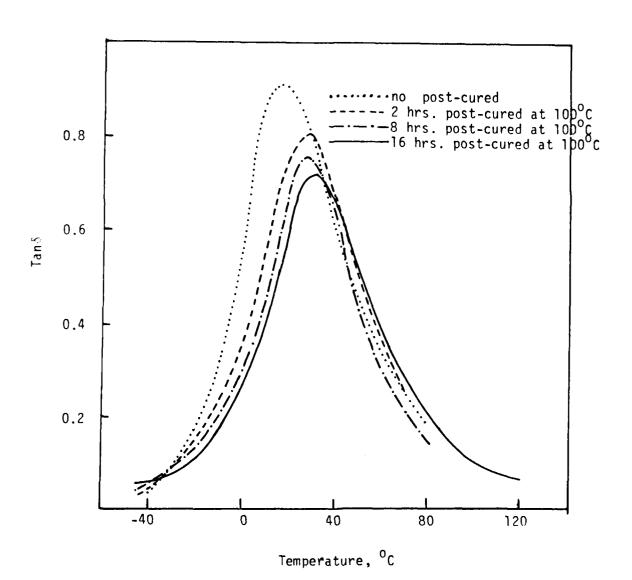
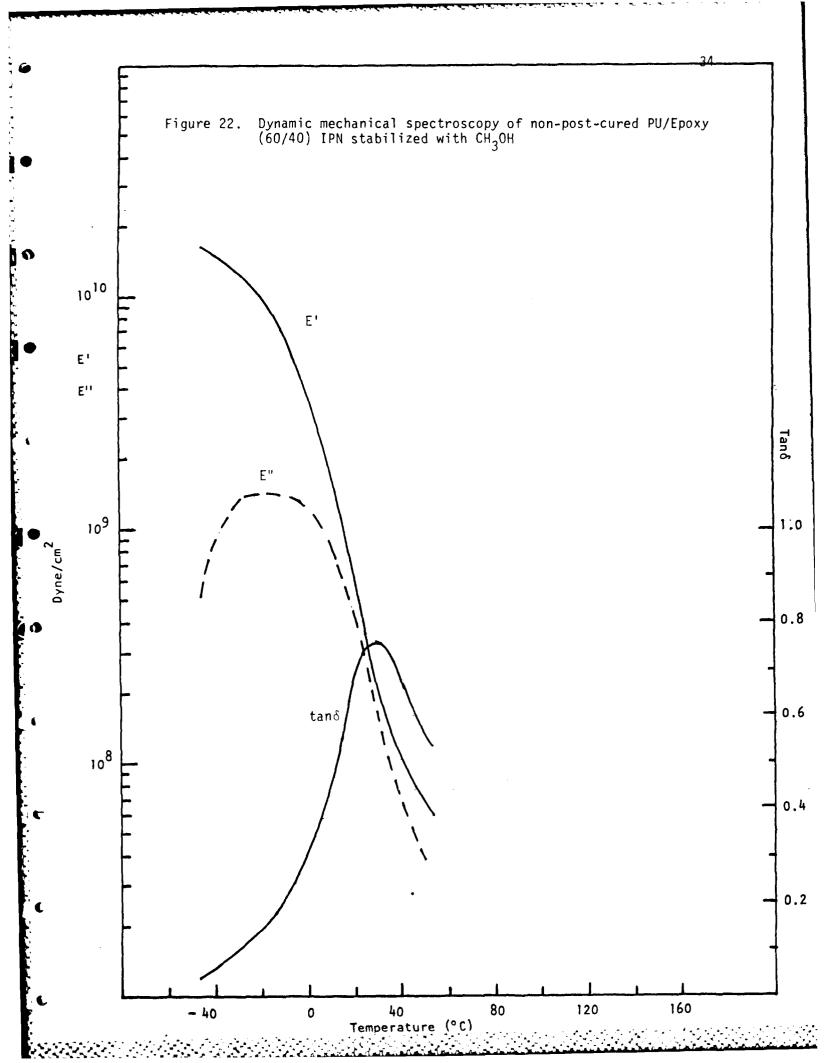


Figure 21. Comparison of the tan δ of samples with different post-curing times





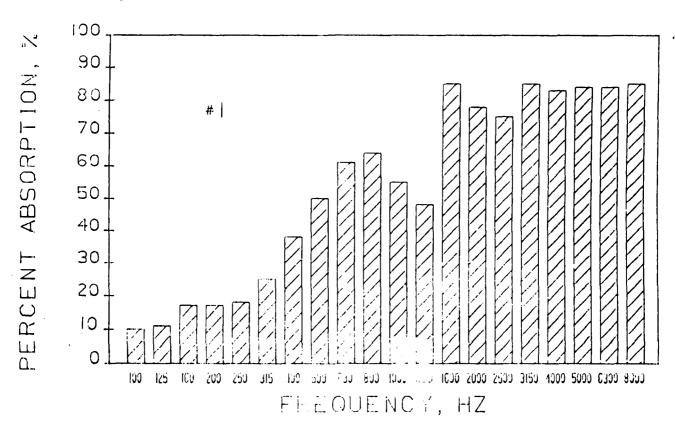


Figure 24. Bottom portion of 70/30 PU/Enoxy IPN foam

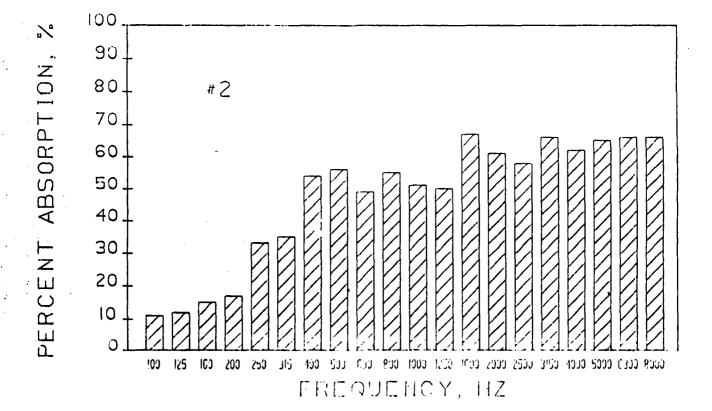


Figure 25. Sound absorption properties of 60/40 PU/Epoxy IPN foam

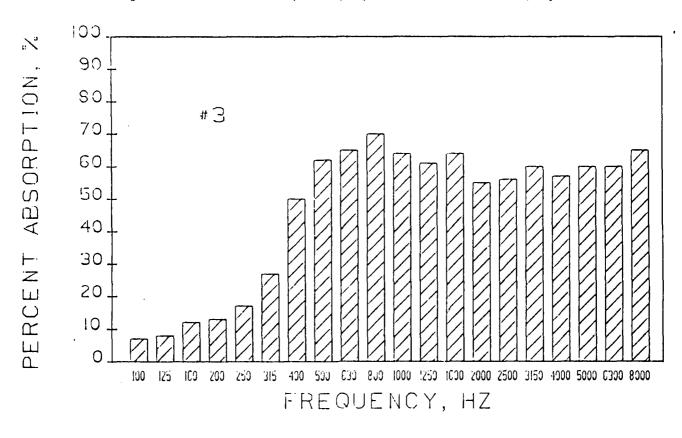
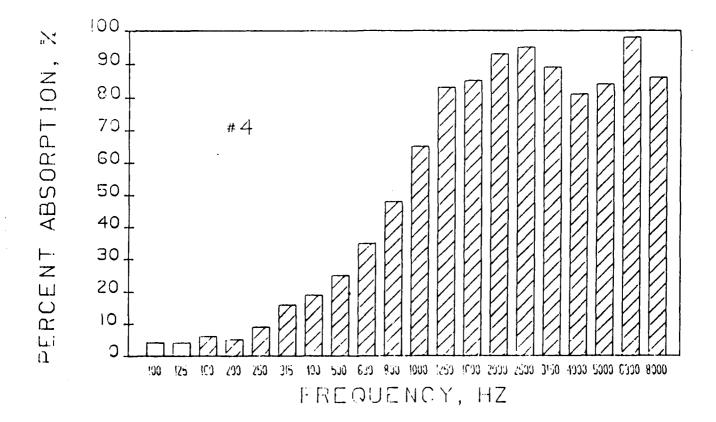


Figure 26. Sound absorption property of 50/50 PU/Epoxy IPN foam



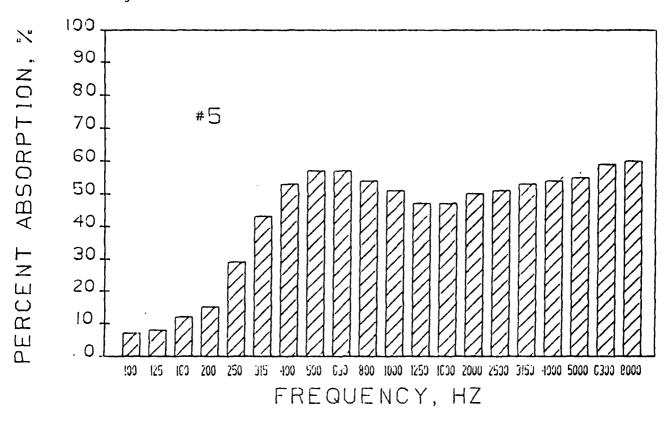
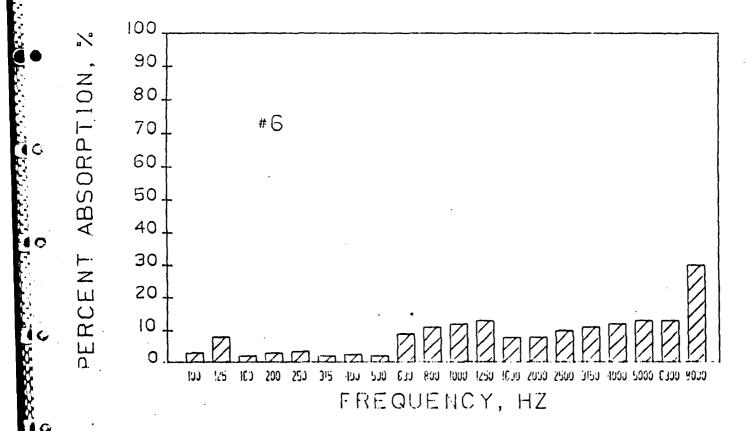


Figure 28. Sound absorption property of 30/70 PU/Epoxy IPN foam



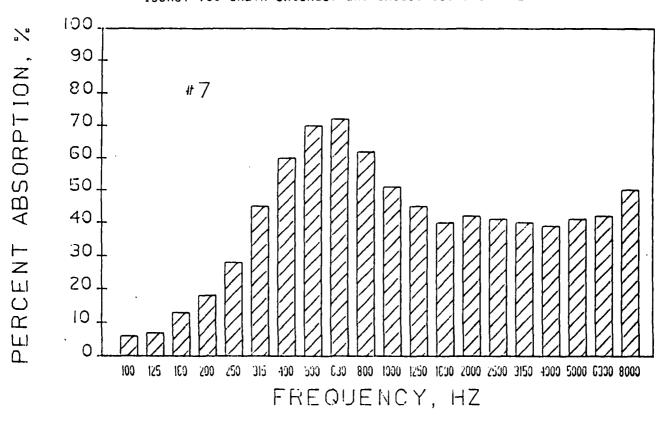
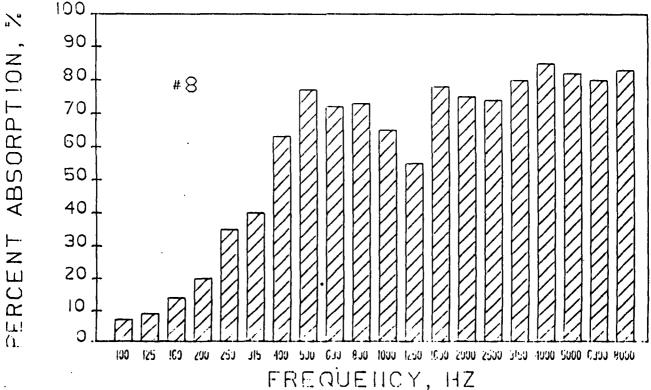
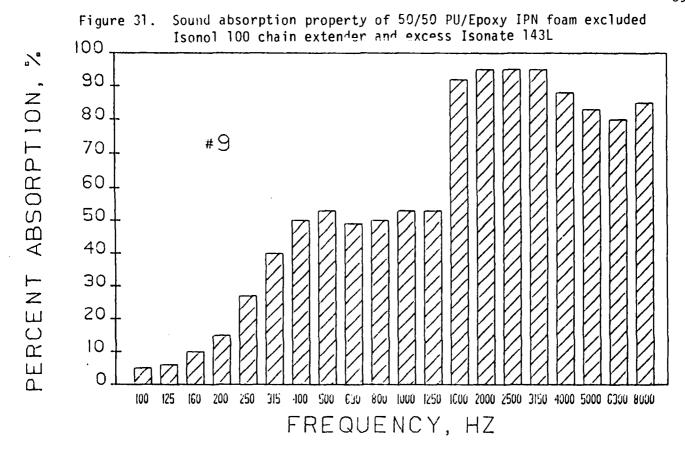
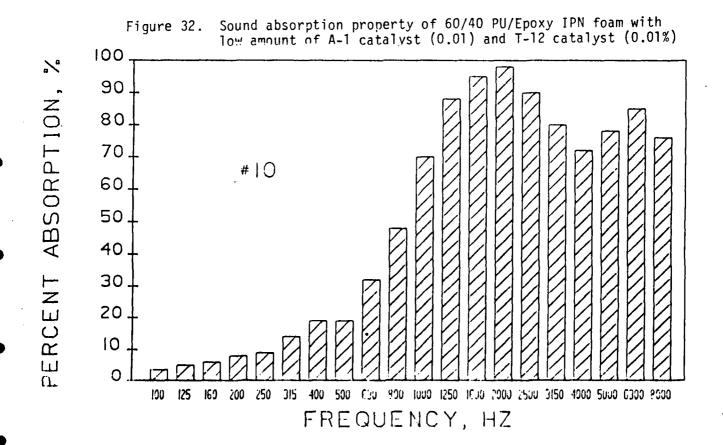
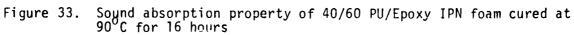


Figure 30. Sound absorption property of 60/40 PU/Epoxy IPN foam excluded Isonol 100 chain extender and excess Isonate 143L









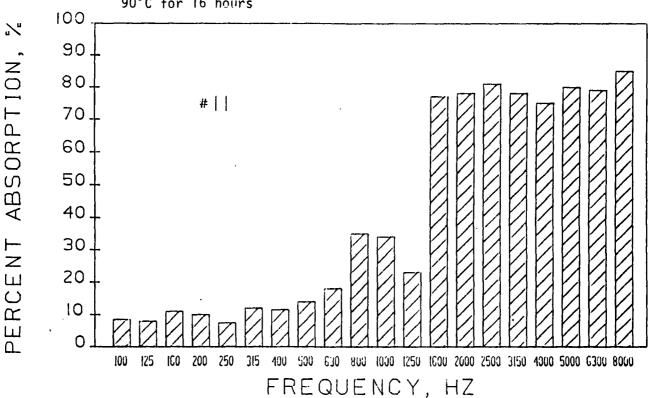
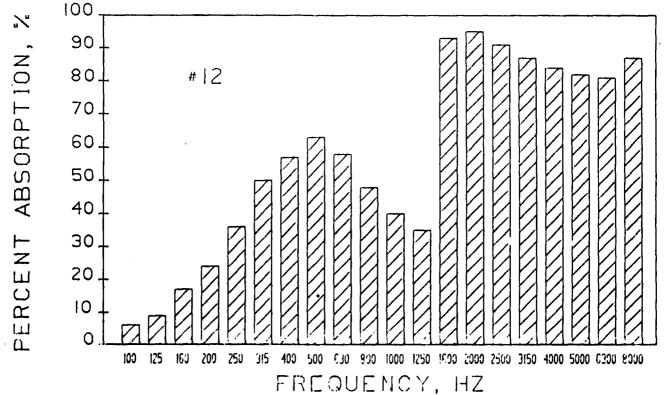
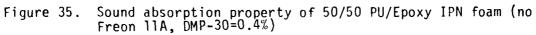


Figure 34. Sound absorption property of 40/60 PU/Epoxy IPN foam without post-curing





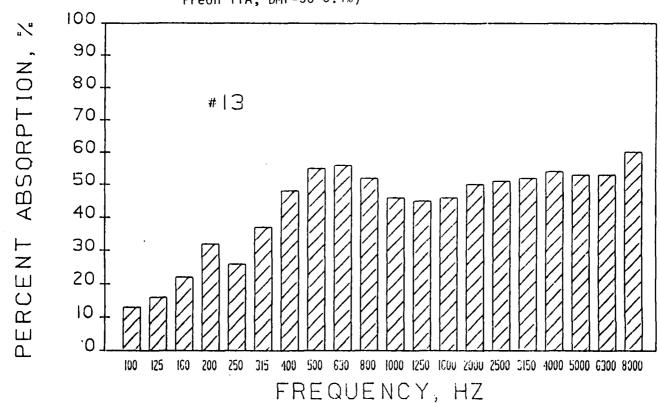
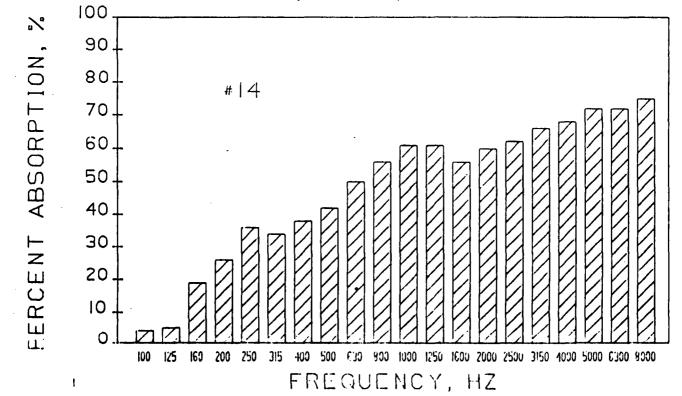
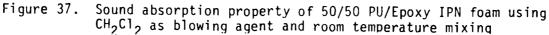


Figure 36. Sound absorption property of 50/50 PU/Epoxy IPN foam (no Freon 11A, DMP-30=1.0%)





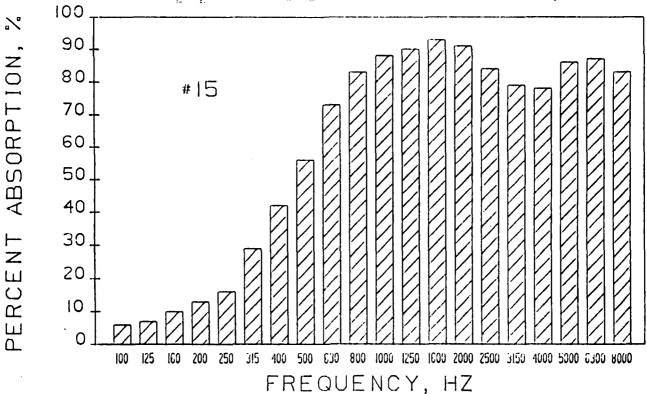


Figure 38. Sound absorption property of 50/50 PU/Epoxy IPN foam using ${\rm CH_2Cl_2}$ as blowing agent, mixing temperature at $80^{\circ}{\rm C}$

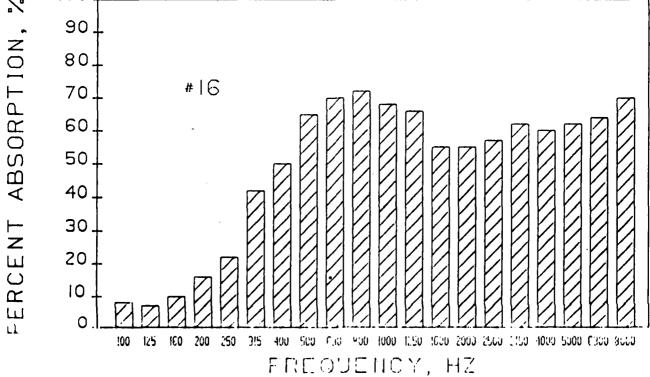


Figure 39. Sound absorption of GM polyurethane foam (Control)

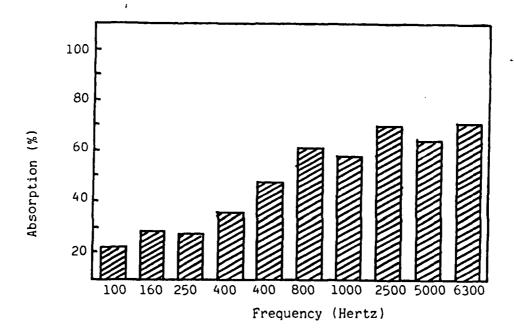
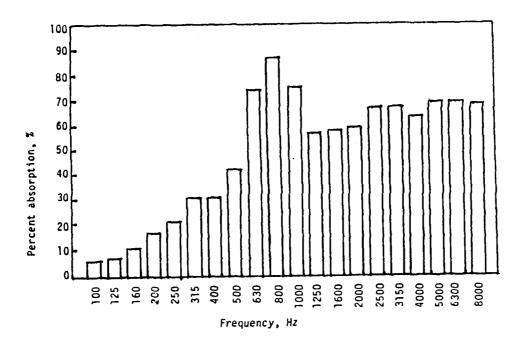


Figure 40. Sound absorption property of University of Detroit's polyurethane foam (Control)



CONTRACTOR OF THE STATE OF THE

Formulation	
Isonate 143L, g	25
Niax 31-28, q	100
Water, g	1.05
A-1, g	0.01
T-12, g	0.06
DC-193, g	0.5
L-540 g	0.5
Freon 11A, g	15
PU/Epoxy	100/0
Water, %	1

DT 1C